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Magnetic characterization of Fe nanoparticles dispersed in phyllosilicate type silicon oxide.

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Abstract. We present the magnetic properties of silica-supported metal (Fe, catalyst) nanoparticles synthesized by precipitation of metal nitrate in ammonia-based medium. Our goal is the study of possible metal-support interactions in the nanoporous catalyst. The temperature dependence of the magnetization for all samples display spin-glass like behavior below c.a. 11-12 K, with clear Curie-Weiss dependence in the high-temperature regime. Spin-glass-like behavior was inferred from dynamic AC susceptibility data after analyzing the frequency-dependence of the in-phase component $\chi'(f)$ by the expression $W = \Delta T_f / [T_f \Delta \log(f)] = 3.0 \times 10^{-3}$. We found that the magnetic behavior of the catalyst is drastically affected by the existence of interactions between the metal and the support.

1. Introduction

Nanosized particles of ferromagnetic metals as Fe, Ni and Co have attracted great interest because of their physical properties and potential applications as catalyst, magnetic recording media and drug delivery systems, etc [1,2].

Numerous physical and chemical methods have been employed to produce metal nanocrystals, including sputtering, grinding, solution phase metal salt reduction, etc. Significant progress has been made in preparing nearly monodisperse metals. Strong magnetic interactions in Ni, Fe and Co particle systems make it difficult to form stable systems. Uncontrolled agglomeration of the magnetic particles often makes it impossible to employ separation procedures which could narrow the size distribution [3,4,].

A favored approach to assemble and maintain metallic nanoparticles is to form nanocomposites whereby the nanoparticles are embedded in a non-magnetic support that provides mechanical and

chemical stability. Oxides such as silica and alumina have been extensively used as support to provide good magnetic insulation [6,7].

2. Experimental details.

The method used for the preparation of the metal based catalyst supported on silica aerosol is a modification of the ammonia method [8]. The procedure was adding silica aerosol for 10% metal in a solution of metal nitrate, to which ammonia solution was added to control the pH about 11 or 5. An excess of ammonia solution was added to dissolve the precipitate. The system was dried in an oven at about 360K for 48 h after stirring for 2 more hours.

Previous characterization studies carried out by XPS, SEM, XRD and FTIR suggested that the metal phyllosilicate is formed by composite sheet in which a layer of octahedrally coordinated cations are sandwiched between two identical layers of linked SiO_4 tetrahedra [9,10]. Dc and ac magnetic susceptibility were performed in a commercial superconducting quantum interference device magnetometer both in zero-field cooling (ZFC) and field-cooling (FC) cycles between 2 and 300K under applied fields up to 5 T. The frequency dependence of both in-phase and out of phase components of the ac magnetic susceptibility was measured by using an excitation field of 2 Oe and driving frequency between 0.1Hz and 1200 Hz.

3. Results and Discussion

The temperature dependence of the ZFC/FC cycles of dc magnetization for Fe particles prepared at pH=5 and 11, measured under applied field $H = 500$ Oe are shown in figure 1 and 2. The curves displayed a clear irreversibility with a sharp peak in the ZFC curves at about $T_p = 12-11$ K. Values of T_p for the different samples are in good agreement with those reported by Gomez-Polo et.al.[6]

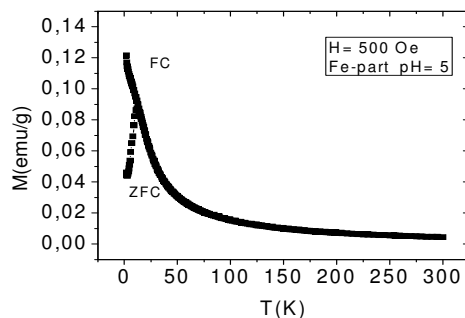


Figure1. ZFC/FC magnetization for Fe particles (pH= 5).

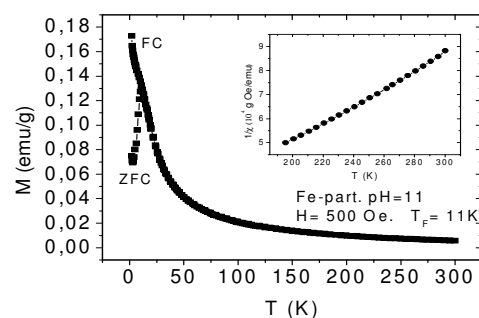


Figure 2. ZFC/FC magnetization for Fe particles (pH= 11) Inset: $1/\chi$ vs T.

No significant changes were obtained at two different pH as is seen in figures 1 and 2. In order to explore the effective interactions in Fe nanosystem we plot χ^{-1} (T) for the high temperature region where the magnetic susceptibility data, follow the Curie-Weiss law with positive paramagnetic Curie temperature $\theta = +54$ K as shown in inset of figure 2, for Fe particles. This result make evident that ferromagnetic interactions are dominant. The variations of magnetization with the static applied magnetic field (up to 5 T at two temperatures, 2K and 300K) are shown in figure 4 and figure 5 for Fe particles prepared at pH =11 and pH = 5. In both cases at 2 K the magnetization increases with increasing applied magnetic field showing a weak ferromagnetism, and tends towards a saturation value $M_s = 3.2 ; 2.0$ (emu/g) for pH= 11 and 5 samples respectively.

The thin, elongated loops in figures 4 and 5 clearly indicate a fairly small coercive field $H_c = 1.45$ kOe for sample pH: 11 and $H_c = 2.0$ kOe for sample pH: 5. But observing figures 4 and 5 we may notice

that the field dependence of the dc magnetization at 300K is a straight line, that means, the Fe system reveals a typical paramagnetic behavior consistent with the high temperature dc magnetization data.

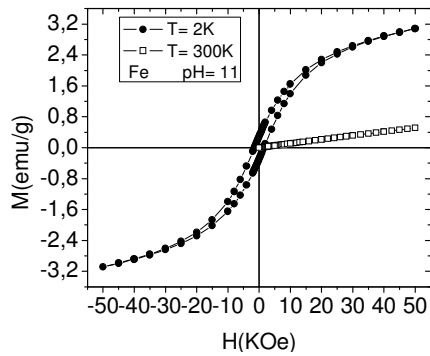


Figure 4. Hysteresis loops for Fe particles (pH:11) at T= 2 K and 300K.

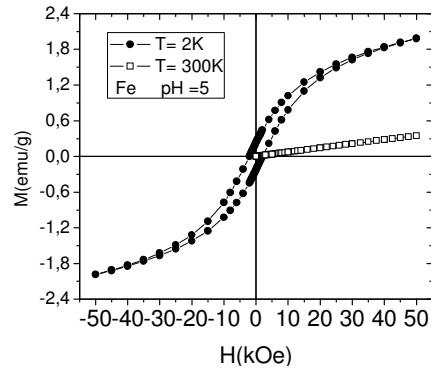


Figure 5. Hysteresis loops for Fe particles (pH:5) at T = 2K and 300K

The existence of a marked splitting in the ZFC-FC curves (see figures 1 and 2) of those types of particles below a certain freezing temperature is characteristic of spin-glass behavior, found in some magnetic spinel compounds and confirm a strong interaction between the metal and the support [11,12].

In order to verify the spin-glass nature of the Fe system the dynamics of the transition was studied through the ac susceptibility. As an example figure 6 and 7 shows the temperature dependence of the ac susceptibility for Fe particles pH=11. The temperature dependence of the in-phase component (χ') obtained at the lowest frequency (0.1 Hz) shows a peak at $T_f \sim 12$ K. The freezing temperature T_f strongly depends on the excitation frequency, as can notice in figure 6. T_f increases with increasing of frequency, as expected for a spin-glass behavior [13,14].

The $\chi_{ac}(T)$ curves display the following basic features of spin-glass systems: (i) the frequency dependent peak shifts towards higher temperatures with the frequency (ii) a decrease in the height of the peak with increasing frequency [11,15]. A quite good criterion to obtain quantitative evidence for spin-glass behavior is the relative shift of the temperature T_f defined as $W = \Delta T_f / [T_f \Delta \log(f)]$. In the presented case for Fe particles, the obtained value was: $W = 3.0 \times 10^{-3}$. This value is smaller than the shift presented in systems with superparamagnetic behavior thus pointing to the the presence of an spin-glass like mechanism [16,17].

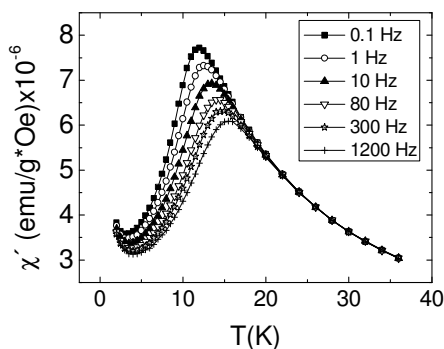


Figure 6. Real component of $\chi_{ac}(f)$ for Fe particles, pH = 11.

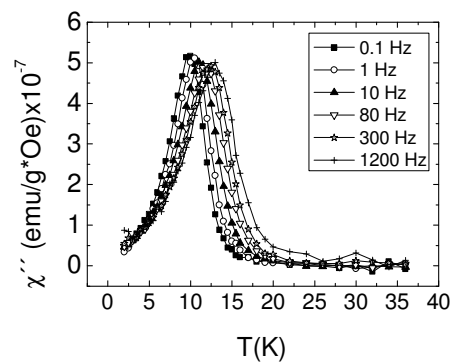


Figure 7. Imaginary part of $\chi_{ac}(f)$ For Fe particles, pH = 11

4. Conclusions

For a spin-glass state to develop, it is suggested that some degree of magnetic frustration is needed from randomness of spins. Therefore we could suggest in our system the spin-glass like behavior could be due to the presence of clusters between particles.

The magnetic characterization of Fe particles supported in phyllosilicate type silicon oxide have shown magnetic interactions in the paramagnetic phase. From ac susceptibility data at different frequencies, a spin-glass like transition was found for Fe nanoparticles system at about $T_f \sim 12$ K. The possible presence of metal-support interactions leads the formation of randomness of spins favoring the spin-glass like behavior due to interacting clusters between particles. Therefore, more experiments of magnetic nature are in progress to elucidate that point.

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